

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of:
Gerhard Meyer et al.

Application Ser. No. 10/539,877

Confirmation No.: 6327

Filed: December 8, 2003

Art Unit: 1794

For: Fire Protection Means and Method for the
Production Thereof

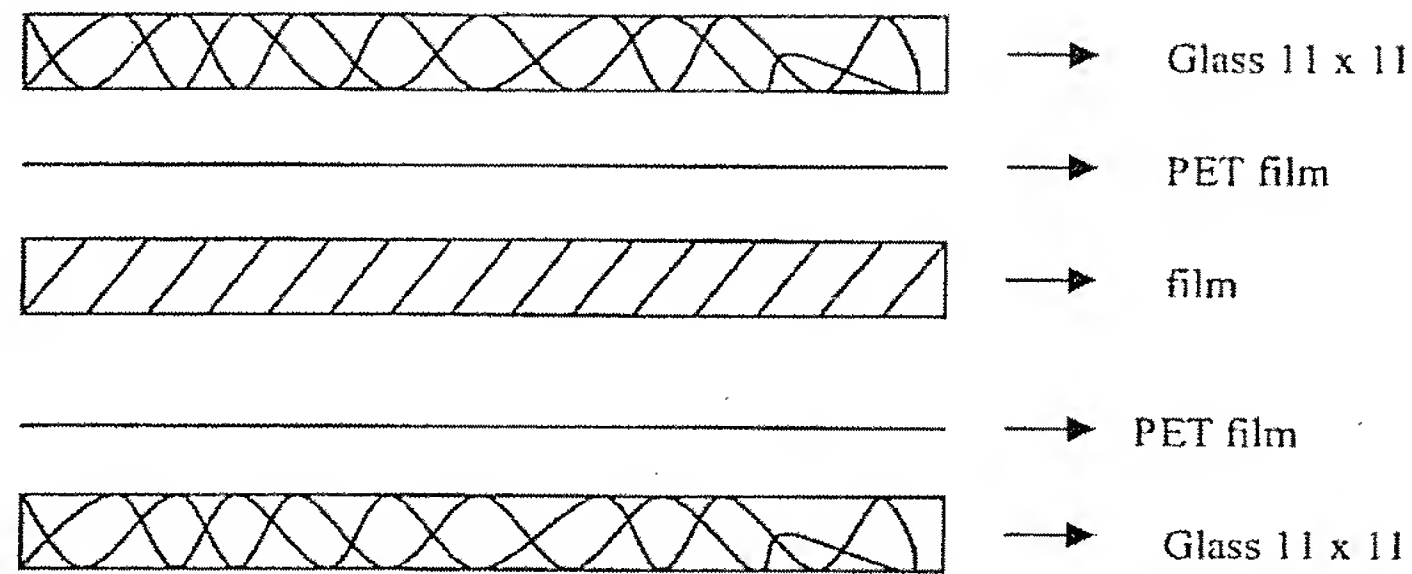
Examiner: Elizabeth A. Robinson

DECLARATION UNDER 37 C.F.R. §1.131

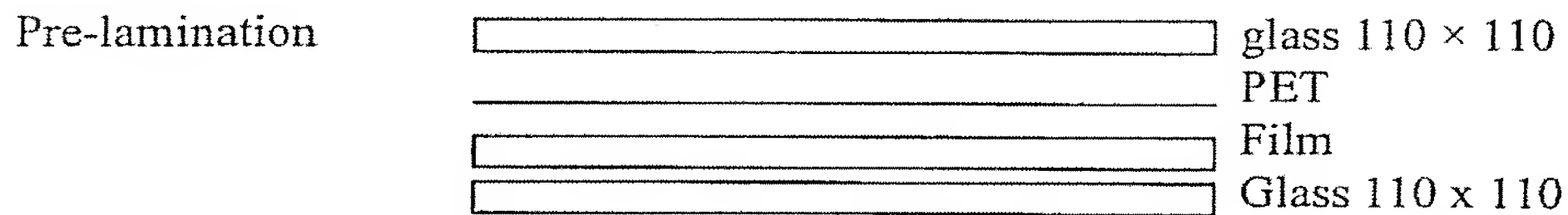
Valentino Villari, hereby states as follows:

1. I am an inventor of the invention described and claimed in the above-identified application, and am personally familiar with the documents attached hereto and the facts recited herein.
2. Attached as an exhibit are copies of documents describing and illustrating preparation of the invention disclosed and claimed in the above-identified application.
3. The dates on the exhibit have been deleted, but each is prior to September 18, 2002.
4. As shown in the exhibit, a fire protection means and fire protection glazing falling within the scope of the invention were prepared before September 18, 2002.
5. More specifically, the fire protection means comprised a hybrid film system, consisting of two or three layers. The two- and three-layered hybrid film system consisted of a film made from a mixture of waterglass and glycerin as intumescent material. This film was covered on one or both flat sides with PET-films (polyethylene-terephthalate) to yield a hybrid film system.

The two- and three-layers comprising hybrid film systems were put in between two sheets of glass, fixed with silicone glue and laminated. As shown in the laboratory notes, the glazing with three-layer hybrid film system had the following composition/build-up:



The fire glazing with a two-layer comprising hybrid system looked as follows:



In addition to the two different layer built-ups of the hybrid film system, the composition of the glycerine/waterglass film used in both cases as organic/ inorganic mixture was varied as shown in the following table to examine its impact on overall glazing properties, especially with respect to haze and transparency:

| | WG | Glycerin calculated | solid material | added | % |
|----|--------|------------------------|-------------------|-----------|------|
| 1% | 127.59 | 1.289 | 37.07 | 1.28 | 1.00 |
| 3% | 127.82 | 3.952 | 38.34 | 3.94 | 3.00 |
| 7% | 124.89 | 9.400 | 40.88 | 9.39 9.40 | 7.00 |
| 9% | 111.57 | 11.034 | 42.15 | 11.11 | 9.00 |

Summarizing the above, fire protection glazings were prepared, consisting of two- or three-layered hybrid film systems laminated between two glass sheets. The hybrid film systems used consisted of an intumescent layer made from glycerine and waterglass in different ratios, i.e. a mixture of an organic and an inorganic component. The second and respectively third film of the hybrid film systems were made from PET, i.e. an organic polyester material, so that the intumenscent layer

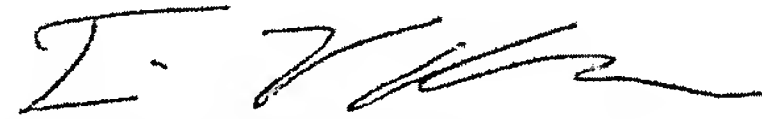
and the PET-layer differed from each other with respect to their components/composition. The glazings prepared were transparent in the visible spectrum. Accordingly, the invention was already made at that time.

6. The acts described above in paragraph 4 and 5 were carried out in the Netherlands.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made punishable by fine or imprisonment, under Section 1001 of Title 18 of the United State Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

01.02.10

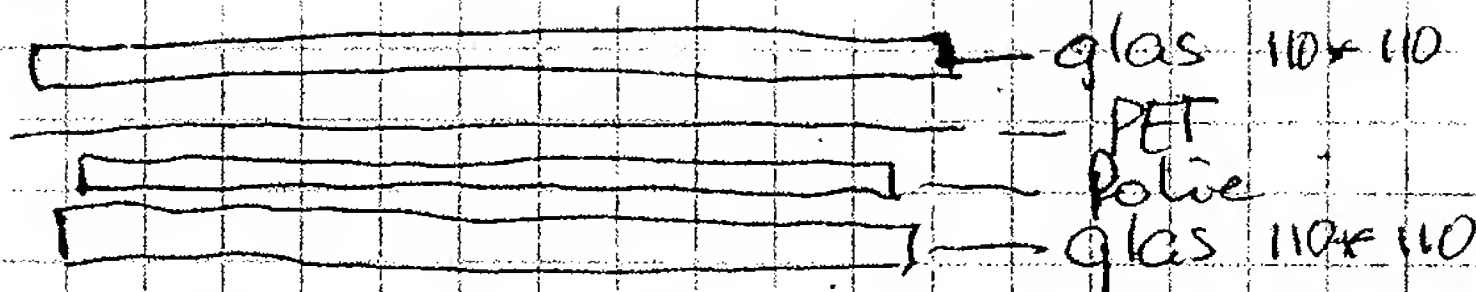
Date



Name: Valentino Villari

EXHIBIT

voorlamineren.



1 * met platgewaakte folie (zie u-10-a). P-A

1 * met "verse folie" van substraat. P-B.

A 2 * glas + 1 * PET + folie P-A. 242,53g.

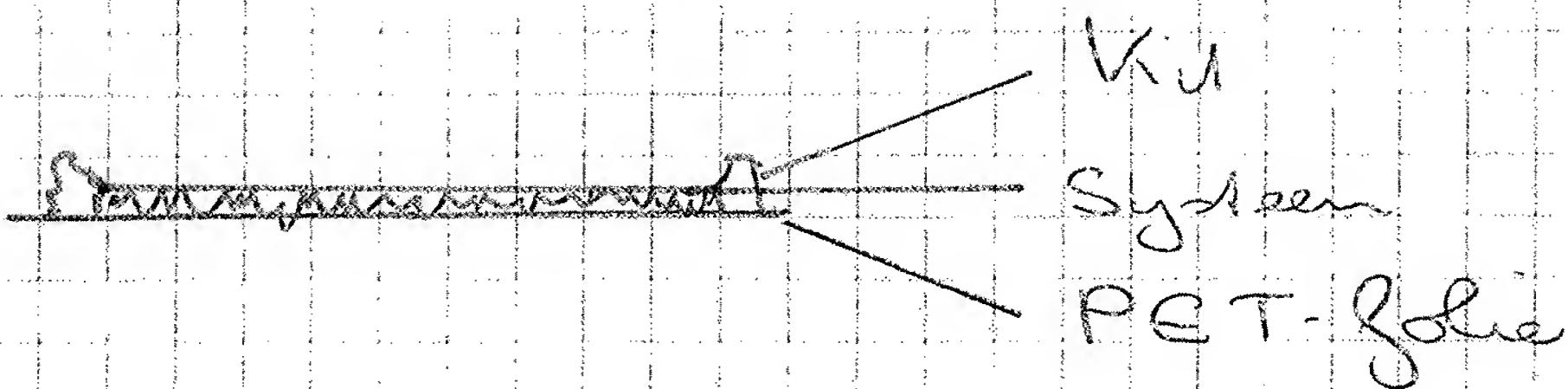
B " " " " P-B. 242,22g.

u-10 in voorverwarmde stoof bij 80°C met 2 * voorverwarmde $\frac{1}{2}$ metalen platen erop weggezet.

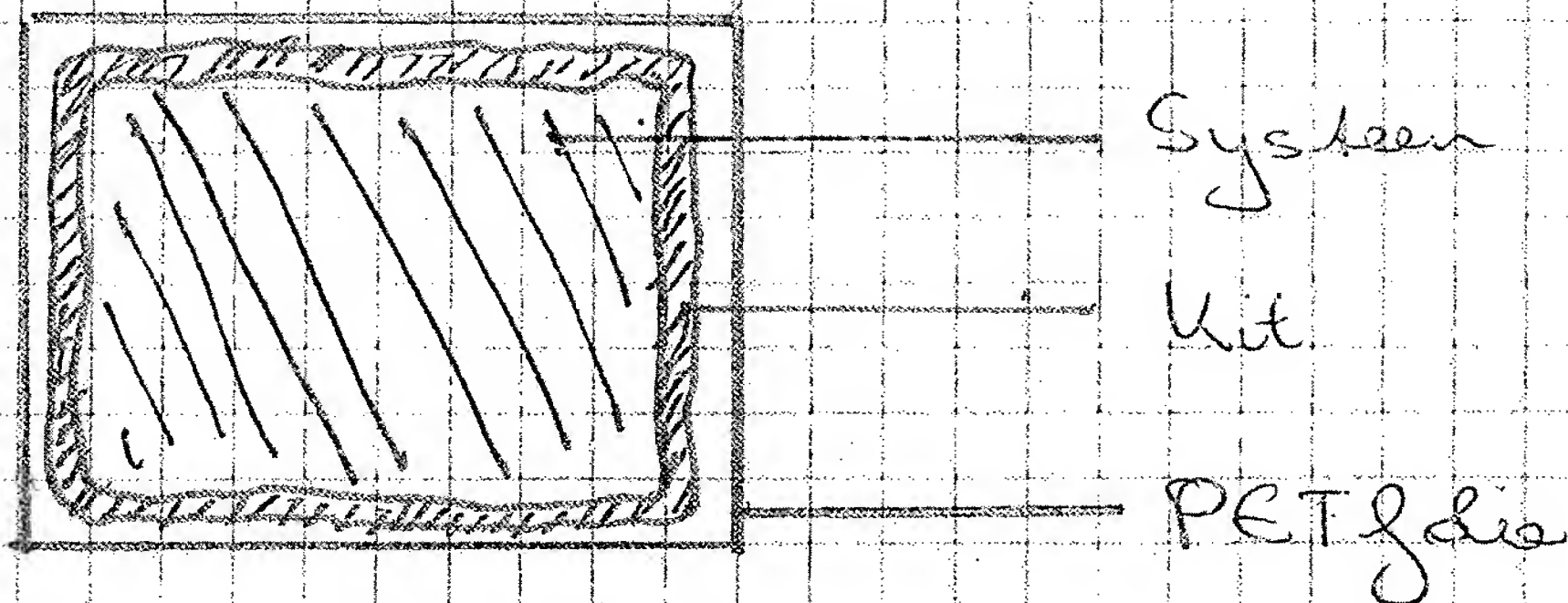
u-6 met glycerine 1, 3, 7, 9 %

| | wb | glycerine berekend | vastestof toegevoegd. | % |
|----|--------|--------------------|----------------------------|------|
| 1% | 127,59 | 1,289 | 37,07 1,28 | 1,00 |
| 3% | 127,82 | 3,952 | 38,34 3,94 | 3,00 |
| 7% | 124,89 | 9,400 | 40,88 9,39 9,40 | 7,00 |
| 9% | 111,57 | 11,034 | 42,15 11,11 | 9,00 |

Zijdenzicht opbouw substraat



bovenaanzicht opbouw substraat



Berekeningen

In de onderstaande tabel staan de resultaten van de berekeningen benodacht.

| Label | Substr. | 130m | 205m | 230m | 265m | 315m | 340m |
|-------|---------|------|-------|------|------|------|------|
| A | glas | 50,7 | 37,10 | 35,6 | - | 30,8 | 29,7 |
| B | glas | 46,8 | 33,8 | 32,7 | 39,8 | 29,1 | - |
| C | glas | 44,3 | 30,3 | 29,4 | 28,0 | - | - |
| d | PET | - | - | 29,7 | 28,3 | - | - |
| E | PET | - | - | 31,6 | - | - | 27,5 |
| F | PET | - | - | 33,7 | - | - | 27,9 |

Restpercentages voor droger.

| Nr | % Na ₂ O | % SiO ₂ | % H ₂ O | % Gly | % Zr-Comp1 | % Zr-Comp2 |
|-----|---------------------|--------------------|--------------------|-------|------------|------------|
| A+d | 7,7 | 25,4 | 61,8 | 5,1 | - | - |
| B+E | 7,8 | 24,5 | 62,6 | 5,0 | 0,7 | - |
| C+F | 8,0 | 24,4 | 62,4 | 4,9 | - | 0,26 |

Na Compleet oxidatie.

| Nr | % Na ₂ O | % SiO ₂ | % ZrO ₂ |
|-----|---------------------|--------------------|--------------------|
| A+d | 23,3% | 76,7% | - |
| B+E | 24,1% | 75,3% | 0,53% |
| C+F | 24,6% | 74,6% | 0,80% |

Folie berekening 60% + 5% glycerine lbr puntbelasting.

VS% 39,61%

| | I | II | III | IV | V | VI |
|-----------------|----------|----------|----------|----------|---------|---------|
| opp substrate | 99,50 | 100,50 | 99,00 | 98,57 | 72,91 | 72,93 |
| massa substrate | 124,7661 | 125,3161 | 125,9719 | 123,8530 | 46,5795 | 46,6204 |
| unwieg berekend | 20,72 | 20,93 | 20,62 | 20,52 | 15,19 | 15,20 |
| unwieg | 20,72 | 20,95 | 20,67 | 20,54 | 15,24 | 15,22 |
| massa bij 25% | 135,71 | 136,38 | 136,89 | 134,70 | 54,47 | 54,23 |

Dikte + Harte XXXX G

| V | | | | | | | | | |
|--------|------|------|------|------|--------|------|------|------|------|
| zonder | met | film | T | H | zonder | met | film | T | H |
| 1,56 | 2,07 | 0,51 | 91,8 | 0,11 | 1,56 | 2,04 | 0,48 | 91,6 | 0,28 |
| 1,56 | 2,07 | 0,51 | 91,4 | 0,20 | 1,57 | 2,03 | 0,46 | 91,7 | 0,18 |
| 1,56 | 2,14 | 0,58 | 91,9 | 0,18 | 1,56 | 2,09 | 0,53 | 91,9 | 0,13 |
| 1,57 | 2,15 | 0,58 | 91,7 | 0,17 | 1,58 | 2,18 | 0,60 | 91,7 | 0,34 |

9.50 O₂ 100 l/h. ~~over aan~~

10.10 over aan


10.35 oven 80 °C

| | I | II | III | IV | V | VI |
|----------------|--------|--------|--------|--------|-------|-------|
| 16.35 (360) | 135,95 | 136,09 | 137,34 | 135,16 | 55,00 | 54,99 |
| 17.35 (420) | 135,77 | 136,52 | 137,08 | 134,88 | 54,80 | 54,77 |
| 18.35 (480) | 135,66 | 136,41 | 136,90 | 134,72 | 54,68 | 54,66 |

lamineren XXXX P-B

glasplaten 100 · 100 voorwarmen op 80 °C

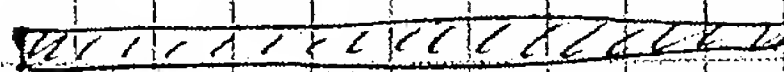
folie 15 minuten in PE ingesaald voorwarmen bij 80 °C

folie tussen de 2 warme glasplaten 4 klemmen erop en terug in de stoof →  !

experiment: Folie plat maken voor lamineren.

 → glas 11 x 11

→ PET folie

 → folie

→ PET folie

 → glas 11 x 11

folie:  PP-C.

totaalgewicht 245,75 g.

drukgewicht: 3003,18 g.

3029,83 g

foto's voor ~~elk~~  PP-C01

 PP-C02

13.30 in stoof bij 80°C

16.00 gewicht 245,71 g.

foto's

17.30

Haze

WG3

WG4

Haze

Trans

Haze

Trans

5,77

85,6

5,94

87,3

23,7

84,6

5,58

87,5

4,29

85,8

1,17

87,9

4 foto's na  PP-C03

 PP-C04

in A4 map onder glas weggelegd.

| 4 massa tijd | massa | % vocht | |
|-----------------|--------|---------|-----------|
| | WG1 | WG2 | WG2 |
| 9.10 | 118.87 | 118.76 | 36.8 21.8 |
| 11.10 | 118.43 | 118.74 | 32.9 21.5 |
| 13.50 | 118.00 | 118.71 | 28.7 21.1 |

punt belasting

| t | m | A | B | C | D |
|---|-------|------|-------------|---|-------------|
| 0 | 206,5 | 0 | 0 | 0 | 0 |
| 1 | +100 | 2,49 | 1,78 | | 2,51 |
| 2 | " | 4,30 | 2,36 | | 3,05 |
| 3 | " | | 2,14 scheur | | 2,20 scheur |
| 4 | " | | | | |

A begint meteen te scheuren.
C ook

lamineren P-PC.

- glas 110 x 110 + siliconen kit.
- glas 110 x 110
- PET
- Film met gebroken randen.
- glas 100 x 100
- glas 110 x 110

12.30 met lijntangen bij 80°C in staaf gezet.

13.45 PET vervangen door 100 x 100 glasplaat.

kg vrf 124 ~~106,12~~ 24,60

1,4-BDO ~~5,50~~ (1,29) 0,46 + 0,84g na oplossen ± 3,5 u
demi 5,59

~~2,98~~
~~1,65~~

1,29
0,46
0,83

vaste stof gehalte 32,59

1,4-BDO : 4,1233 g

ENGLISH TRANSLATION OF EXHIBIT

Lamination

Pre-lamination

| | |
|--|-----------------|
| | glass 110 × 110 |
| | PET |
| | Film |
| | Glass 110 x 110 |

1 × with flattened film (see [REDACTED]) [REDACTED] P-A

1 × with “fresh film” of substrate 011001P-B

A 2 × glass + 1 + PET + film [REDACTED] P-A 242.53 g

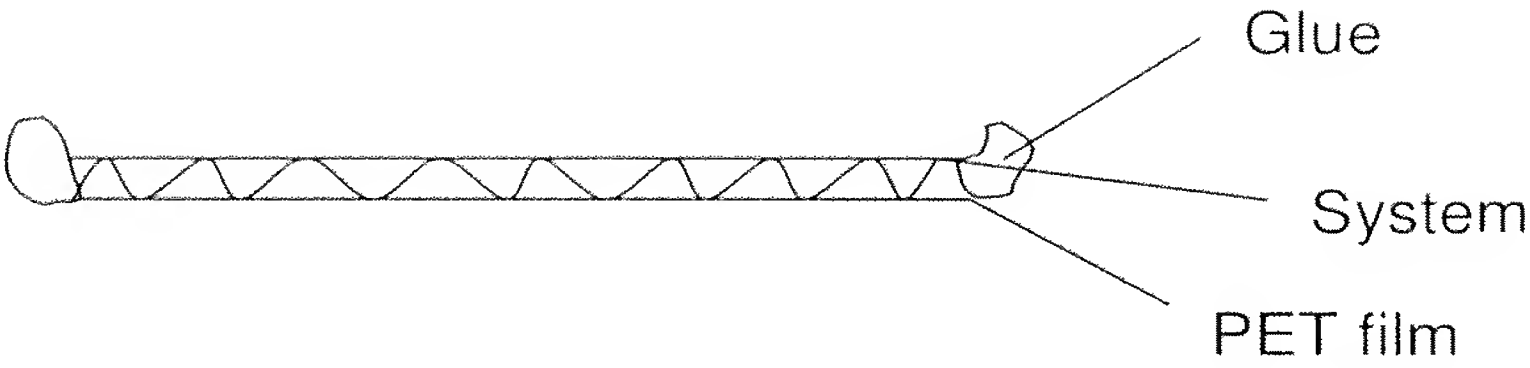
B " " " " " [REDACTED] P-B 242.22 g

No. 10 in pre-heated kiln at 80 °C [176 °F] put away with 2 x pre-heated metal plates on it

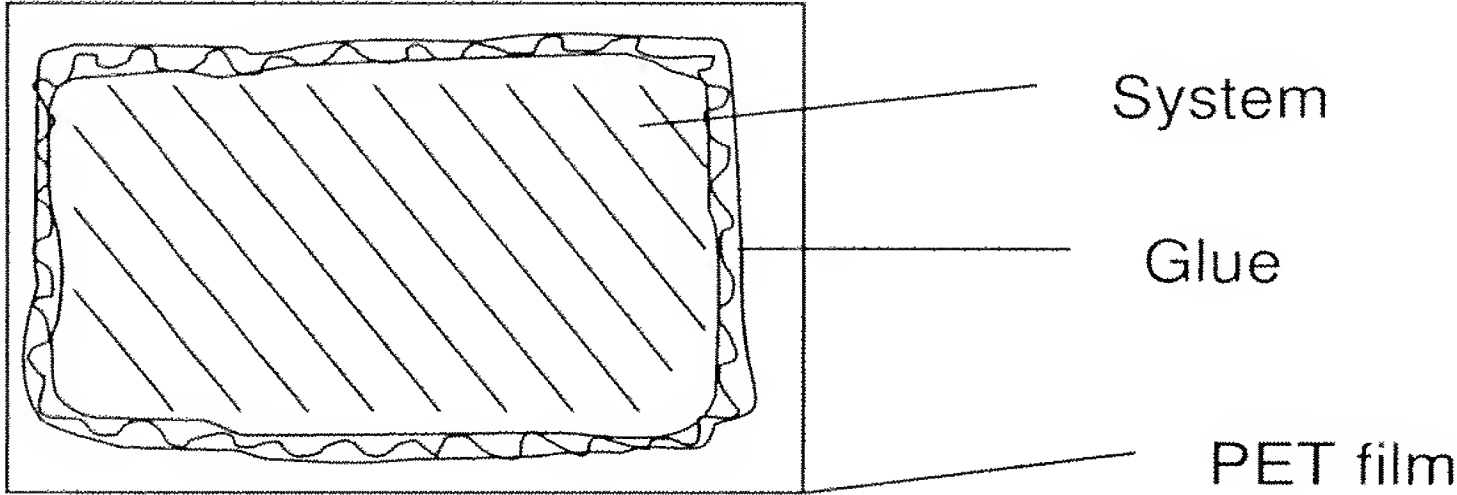
WG (waterglass) with glycerin 1%, 3%, 7%, 9%

| | WG | glycerin calculated | solid material | added | % |
|----|--------|------------------------|-------------------|----------------------|------|
| 1% | 127.59 | 1.289 | 37.07 | 1.28 | 1.00 |
| 3% | 127.82 | 3.952 | 38.34 | 3.94 | 3.00 |
| 7% | 124.89 | 9.400 | 40.88 | 9.39 9.40 | 7.00 |
| 9% | 111.57 | 11.034 | 42.15 | 11.11 | 9.00 |

Side view of substrate structure



Top view of substrate structure



Calculations

The table below shows the results of the core moisture calculations

| Dish | Substrate | 130 m | 205 m | 230 m | 265 m | 315 m | 340 m |
|------|-----------|-------|-------|-------|-------|-------|-------|
| A | glass | 50.7 | 37.10 | 35.6 | | 30.8 | 29.7 |
| B | glass | 46.8 | 33.8 | 32.7 | 39.8 | 29.1 | - |
| C | glass | 44.3 | 30.3 | 29.4 | 28.0 | - | - |
| D | PET | - | - | 29.7 | 28.3 | - | - |
| E | PET | - | - | 31.6 | - | - | 27.5 |
| F | PET | - | - | 33.7 | - | - | 27.9 |

Remaining percentages before drying

| No. | % Na ₂ O | % SiO ₂ | % H ₂ O | % glyc. | % Zr Compl. 1 | % Zr Compl. 2 |
|-------|---------------------|--------------------|--------------------|---------|------------------|------------------|
| A + D | 7.7 | 25.4 | 61.8 | 5.1 | - | - |
| B + E | 7.8 | 24.5 | 62.6 | 5.0 | 0.17 | - |
| C + F | 8.0 | 24.4 | 62.4 | 4.9 | - | 0.26 |

After complete oxidation

| No. | % Na ₂ O | % SiO ₂ | % ZrO ₂ |
|-------|---------------------|--------------------|--------------------|
| A + D | 23.3% | 76.7% | - |
| B + E | 24.1% | 75.3% | 0.53% |
| C + F | 24.6% | 74.6% | 0.80% |

Film preparation WG + 5% glycerin for purposes of point loading

| VS% 39.61% | I | II | III | IV | V | VI |
|--------------------------------|----------|----------|----------|----------|---------|---------|
| substrate surface | 99.50 | 100.50 | 99.00 | 98.51 | 72.91 | 72.99 |
| substrate weight | 124.7661 | 125.3161 | 125.9719 | 123.8530 | 46.5795 | 46.6204 |
| calculated weighed-in quantity | 20.72 | 20.93 | 20.62 | 20.52 | 15.19 | 15.20 |
| weighed-in quantity | 20.72 | 20.95 | 20.67 | 20.54 | 15.24 | 15.22 |
| weight at 25% | 135.71 | 136.38 | 136.89 | 134.70 | 54.47 | 54.23 |

Thickness + haze XXXXXXXXXX G
V

| without | with | film | T | H | without | with | film | T | H |
|---------|------|------|------|------|---------|------|------|------|------|
| 1.56 | 2.07 | 0.51 | 91.8 | 0.11 | 1.56 | 2.04 | 0.48 | 91.6 | 0.28 |
| 1.56 | 2.07 | 0.51 | 91.4 | 0.20 | 1.57 | 2.03 | 0.46 | 91.7 | 0.18 |
| 1.56 | 2.14 | 0.58 | 91.9 | 0.18 | 1.56 | 2.09 | 0.53 | 91.9 | 0.13 |
| 1.57 | 2.15 | 0.58 | 91.7 | 0.17 | 1.58 | 2.18 | 0.60 | 91.7 | 0.34 |

9:50 a.m. O₂ 400 l/h even on
10:10 a.m. oven on
10:35 a.m. oven 80 °C [176 °F]

| | I | II | III | IV | V | VI |
|--------------------|--------|--------|--------|--------|-------|-------|
| 4:35 p.m. (360) | 135.95 | 136.69 | 137.34 | 135.16 | 55.00 | 54.99 |
| 5:35 p.m. (420) | 135.76 | 136.52 | 137.08 | 134.88 | 54.80 | 54.77 |
| 6:35 p.m. (480) | 135.66 | 136.41 | 136.90 | 134.72 | 54.68 | 54.66 |

Lamination XXXXXXXXXX P-B

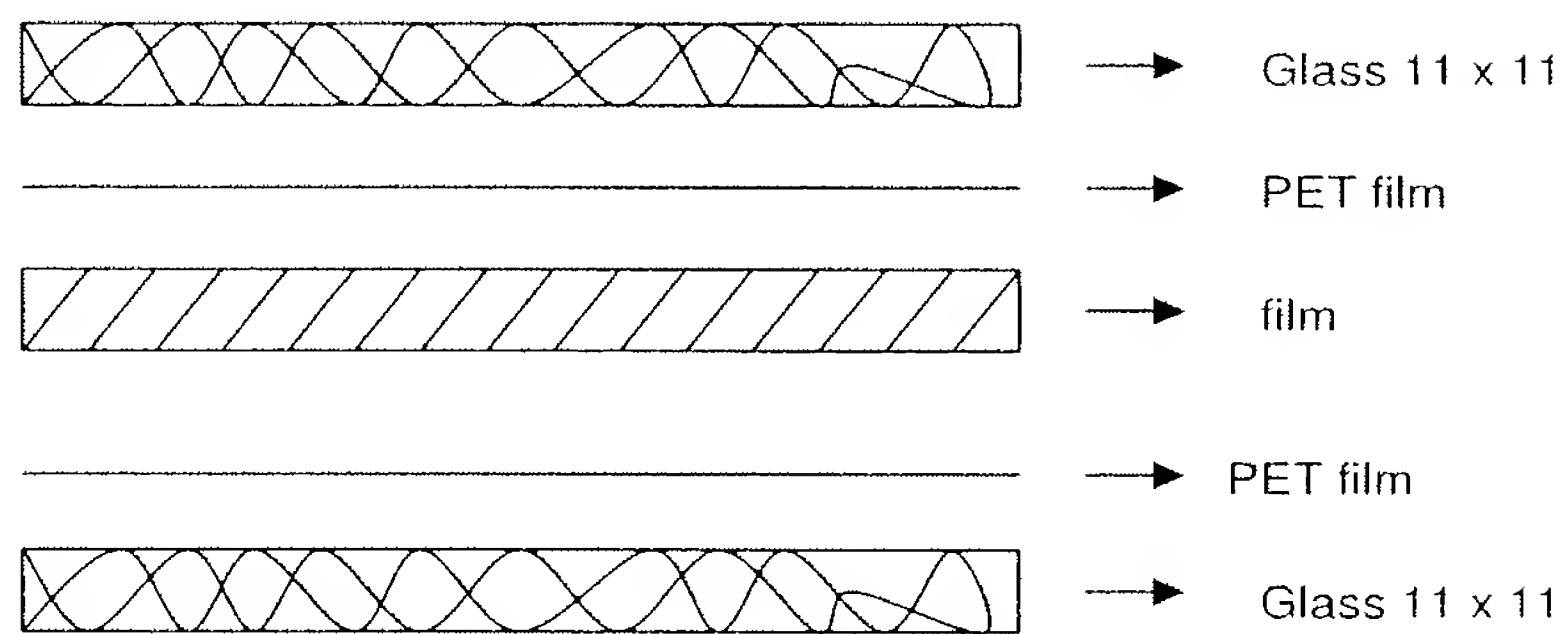
Glass plates 100 × 100 pre-heated to 80 °C [176 °F]

Film sealed in PE for 15 minutes, pre-heated to 80 °C [176 °F]

Film between the two hot glass plates, 4 clamps on them and back into the kiln

⇒👍!

Experiment: flattening the film prior to lamination



Film XXXXXXXXXX PP-C
total weight: 245.75 g
pressure weight: 3003.18 g
3029.83 g
Photos for XXXXXXXXXX PP-C01
XXXXXXXXXX PP-C02

1:30 p.m. into the kiln at 80 °C [176 °F]

4:00 p.m. weight 245.71 g
photos

5:30 p.m.

| WG3 | | WG4 | |
|------|--------|------|--------|
| Haze | Trans. | Haze | Trans. |
| 5.77 | 85.6 | 5.94 | 87.3 |
| 23.7 | 84.6 | 5.58 | 87.5 |
| 4.29 | 85.8 | 1.17 | 87.9 |

Ok photos after XXXXXXXXXX PP-C03
XXXXXXXXXX PP-C04

Filed in A4 folder under “glass”

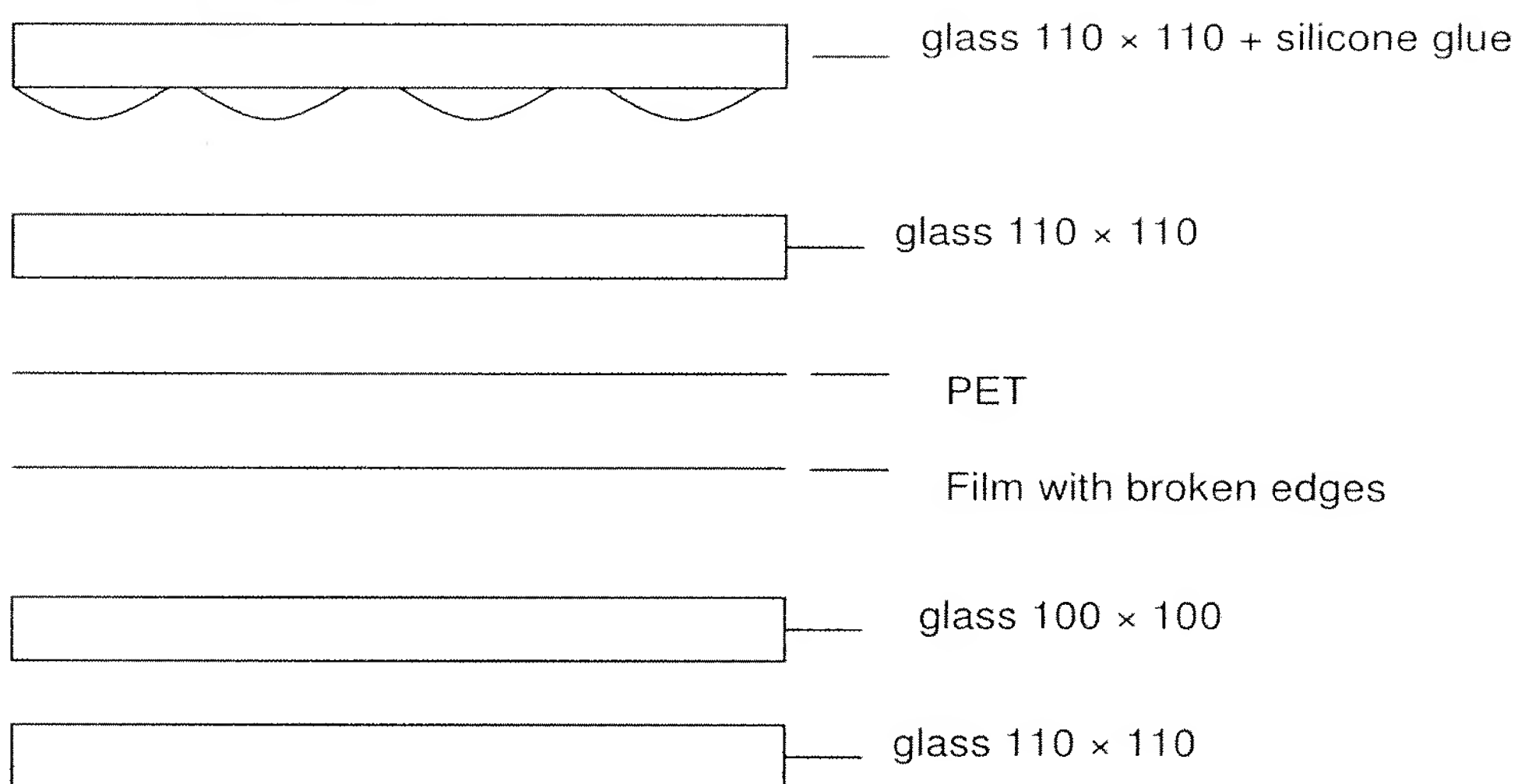
| | | Weight | | % Moisture | |
|--------|------------|--------|--------|------------|------|
| weight | Time | WG1 | WG2 | WG1 | WG2 |
| | 9:10 a.m. | 118.87 | 118.76 | 36.8 | 21.8 |
| | 11:10 a.m. | 118.43 | 118.74 | 32.9 | 21.5 |
| | 13:50 a.m. | 118.00 | 118.71 | 28.7 | 21.1 |

Point loading [redacted]

| T (time) | M (mass) | A↑ | B↑ | C↓ | D↓ |
|----------|----------|-----------|------|----|-----------|
| 0 | 206.5 | 0 | 0 | | 0 |
| 1 | + 100 | 2.49 | 1.78 | | 2.51 |
| 2 | " | 4.30 | 2.36 | | 3.05 |
| 3 | " | 2.14 tear | | | 2.20 tear |
| 4 | " | | | | |

A begins to tear immediately
C as well

Lamination [redacted] P-C



12:30 p.m. placed into the kiln at 80°C [176°F] with clamping tool

1:45 p.m. PET replaced with 100 × 100 glass plate

| | | |
|-----------------------------|-------------------|---|
| WG ref. 124 | 106.12 | 24.60 |
| 1.6-BDO (5.58) | (1.29) | 0.46 + 0.84 g after dissolving ± 3.5 hrs. |
| demi (demi-water) | | 5.59 |

[illegible]

~~1.59~~
0.60
0.88

Solid material content 32.59

1.4-BDO 4.1283%